Federal Democratic Republic of Ethiopia

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ET 1203 (2005) (English): Beeswax - Specification

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ETHIOPIAN
STANDARD

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Beeswax — Specification

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Descriptors: agricultural products, beeswax, chemical requirements labeling packing sampling analysis,

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Foreword

This Ethiopian Standard has been prepared under the direction of the Agriculture and Food Technology Technical Committee and published by the Quality and Standards Authority of Ethiopia (QSAE).

In the preparation of this Ethiopian Standard references have been made to the following:

- IS 1504-1974 Specification for beeswax
- British Pharmacopoeia 1980

Acknowledgement has been made for the use of information from the above publication.

Bees wax- Specifcation

1 Scope

This Ethiopian Standard specifies requirements and method of sampling and test for beeswaxes

2 Definition

2.1

beeswax

shall mean the building material of the honeybee. It is produced from four pairs of glands situated under the abdomen of the worker bee. The bees produce it when they are gathering nectar and are used to build the honeycomb in which the nectar is stored.

3 Types of beeswax

3.1 The commercial beeswax shall be of the following two types

a) Crude beeswax – the material obtained from the honeycombs after the removal of honey and after subjecting the material to a preliminary treatment, such as melting, Cumming, decantation and moulding.
It shall not contain any other added material such as paraffin wax, starch and animal tallow.

b) Refined bees wax- The material obtained after subjecting crude beeswax to further purification by melting and finer filtration

4 Requirements

4.1 The material shall be in the form as agreed to between the purchaser and the supplier. In the absence of such an agreement it shall be in the form of slabs

4.2 The color of refined beeswax shall be whitish yellow to yellowish brown and it shall have a characteristic aroma

4.3 Beeswax shall be insoluble in water and resistant to many acids, but soluble in most organic solvents such as ether, benzene, chloroform, turpentine oil and after warming in alcohol

4.4 The material shall comply with the requirements given in the following table.

Table I showing requirements of beeswax

Ser. N0	Characteristics	Requirements		Test methods
		Crude beeswax	Refined beeswax	
1	Specific gravity at 20 ^{0}C	0.9500-9800	0.9550-0.9800	Annex A
2	Melting point, ^{0}C	58-64	61-66	Annex B
3	Refractive index at 75 ^{0}C	1.4400-1.4450	1.4400-1.4450	Annex C
4	Ash, % by mass max.	0.60	0.20	Annex D
5	Total volatile matter, % by mass, max.	1.0	0.75	Annex E
6	Acid value, max.	17-24	17-24	Annex F
7	Saponification value min.	80.0	85-105	Annex G
8	Ester value	63-84	70-80	Annex H
9	Fats and Fatty acids	To pass the test	To pass the test	Annex J
10	Paraffin and other waxes	To pass the test	To pass the test	Annex K

5 Packing and labeling

5.1 Packing

5.1.1 Unless otherwise agreed to between the purchaser and the supplier, each slab shall be wrapped in greaseproof paper or a suitable plastic material like polyethylene. A number of such slabs shall be packed together in a suitable and clean container.

5.2 Libeling

5.2.1 each container shall be suitably marked so as to give the following information

i Name and type of the material
ii Name of the supplier
iii Batch number or code number
iv Net weight
v Country of origin

6 Sampling

6.1 In drawing, preparing storing and handling samples, the following precautions and direction shall be considered

6.1.1. Samples shall be taken in a protected place not exposed to damp air, dust or soot

6.1.2 The sampling instrument shall be clean and dry when used.

6.1.3 Precautions shall be taken to protect the samples, the material being, the sampling instrument and the containers for the sample from adventitious contamination.

6.1.4 The sample shall be placed in clean and dry glass containers. The sample containers shall be of such size that they are all most completely filled by the sample.

6.1.5 Each container shall be sealed air- tight after filling and marked with full detailed of sampling, batch or code number, name of the manufacturer, and other important particulars of the consignment.

6.1.6 Sample shall be stored in such a manner that the temperature of the material does not vary from the normal temperature.

6.2 Scale of sampling

6.2.1 Lot All the containers in a single consignment of the same material drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches of manufacture, the batch shall marked separately and the groups of containers in each batch shall constitute a separate lot.

6.2.2 Sample shall be tested for each lot for ascertaining the conformity of the material to the requirement of the specification

6.2.3 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with the table shown below and the containers shall be selected at random from the lot.

Table II showing number of containers to be selected for sampling

Lot size	Number of containers to be selected
Up to 25	3
26 to 100	4
101 to 500	5
501to 1000	7
1001and above	9

6.3 Preparation of individual sample

6.3.1 Draw with suitable sampling instrument equal quantities of the material from different parts of the container till about 500g of the material drawn.

6.3.2 divide the sample into three equal parts. Each part so obtained shall constitute an individual sample representing the container and shall be transferred immediately to thoroughly clean and dry containers and sealed airtight and marked with particulars of the consignment.

6.3.3 The individual samples so obtained shall be divided into three sets in such a way that each set has a sample representing each selected container. One of these shall be marked for the supplier another for the laboratory and the third for the referee.

6.4 Preparation of composite sample

6.4.1 From the material from each of the selected container, remaining after the individual sample has been taken, approximately equal quantities of the material shall be taken and mixed together so as to from a composite sample weighing about 150 gm. This composite sample shall be divided into three equal parts and transferred to clean and dry containers sealed air –tight and labelled with particulars of the consignment. One of these composite samples shall be for the supplier, another for the laboratory and the third for the referee.

6.5 Referee sample

6.5.1 Referee sample shall consist of a set of individual and a composite sample marked for this purpose and shall bear the seals of the supplier and the signature of the sampler. These shall be kept at appropriate place.

6.6 Number of tests

6.6.1 Tests for description, melting point, total volatile matter and ash shall be conducted on each of the samples containing a set of individual samples.

6.6.2 Tests for specific gravity, refractive index, acid value saponification value; fats tatty acid, paraffin and other waxes shall be conducted on the composite sample.

6.6.3 A lot shall be declared to have satisfied all the requirements of the specifications given in section 4

7 Marking

7.1 Each container of beeswax that is in compliance with this Ethiopian Standard shall bear the quality mark upon approval by the concerned Authority

8 Compliance

8.1 A lot of beeswax shall be deemed to comply with this Ethiopian Standard if upon inspection and testing the provision under clause 4, and 5, are satisfied.

Annex A
(normative)

Determination of specific gravity

A1 Apparatus

A1.1 Water-Bath - maintained at 20 ± 1°C.
A1.2 Specific Gravity Bottle- 25 ml capacity.

A2 Reagent

A2.1 Alcohol

A3 Procedure

A2.1 Melt approximately 2 g of the material in a porcelain crucible at a temperature of about 100°C.

A2.2 Allow to cool to room temperature. Remove the solidified beeswax from the crucible, warming slightly if necessary.

A2.3 Attach a tarred silk or similar item thread that will suspend the beeswax during weighing.

A2.4 Store the sample for 2 hours at a temperature of 20 ± 1°C. Determine the mass of the sample, first in air and then in rectified spirit maintained at 20± 1°C. Determine the specific gravity at 20°C/20°C of the rectified spirit by means of the specific gravity bottle.

A4 Calculation

Specific gravity at 20°C/20°C= $\frac{M1d}{M1 - M2}$

Where,
M1 mass in g of the material in air,
d specific gravity of rectified spirit, and
M2 mass in g of the material in alcohol.

Annex B

(normative)

Determination of melting point

B1 Apparatus

B1.1 Thermometer-*of* a suitable type, with an accuracy of 0. 1°C and graduated at every 0.1°C.

B1.2 *Test-Tube-with a* centrally bored cork to take the thermometer. The cork shall have a slit so as to permit circulation of air.

B1.3 *Water-Bath*- of a suitable type, with a thermometer.

B2 Procedure

B2.1 Melt the material by warming it in a water-bath at a temperature just sufficient to melt it.

B2.2 Dip the thermometer and withdraw, so as to get the bulb thinly coated with the wax. Let it stand for 24 hours.

B2.3 Insert this thermometer into the test-tube through the bored cork and then place the test-tube in the water-bath.

B2.4 Raise the temperature gradually, at the rate of 1°C in 3 minutes. Note the temperature, accurately to 0 .1 °C, at which a transparent drop forms on the end of the thermometer bulb. Record this temperature as the melting point of the material.

Annex C
(normative)

Determination of refractive index

C1 Apparatus

C1.1 Refractometer

C1.2 Water bath thermostatically controlled and maintained at $75 \pm 1\,^{0}C$

C2 Procedure

C2.1 The sample shall be melted and filtered through fast filter paper to remove any impurities and last traces of moisture.

C2.2 The temperature of the refractometer shall be adjusted at $75 \pm 1\,^{0}C$by circulating water from the water bath.

C2.3 Place few drops of the sample on the lower prism The prism shall be closed tightened firmly allowed to stand for one or two minutes

C2.4 Read and record the refractive index after the sample has attained the test temperature.

Annex D

(normative)

Determination of ash

D1 Apparatus

D1.1 Platinum porcelain, or silica *Dish* - having a capacity of 100 ml.

D2 Procedure

D2.1 Heat the platinum dish to redness, cool to room temperature in a desiccator and weigh. Take about 50 g of the material in a watch-glass and weigh accurately.

D2.2 Transfer about three-quarters of this quantity to the platinum dish and heat on a Bunsen burner so that the material burns gently at the surface. When about half of the material is burnt away, stop heating, cool and add the remainder of the material.

D2.3 Weigh the watch-glass again and find, by difference, the exact mass of sample transferred to the platinum dish. Heat again as before till the material is completely charred.

D2.4 Incinerate in a muffle furnace at 550°C to 650°C for 1 hour. Cool to room temperature in a desiccator and weigh. Repeat incineration, cooling and weighing until the difference between two successive weightings is less than one milligram:

D3 Calculation

Ash, percent by mass $= \frac{100M2}{M1}$

Where,
M2= mass in g of the ash, and
MI = mass in g of the material taken for the test.

Annex E
(normative)

Determination of Total volatile matter

E1 Apparatus

E1.1 Oven maintained at 105 ^{0}C
E1.2 Analytical balance
E1.3 Metal or aluminium dish

E2 Procedure

E2.1 Weigh accurately about 10 g of the material in a suitable dish ,previously dried and weighed ,and place it in an oven maintained at 105 2 ^{0}C for 6 hours.

E2.2 Cool the dish in a desiccator and weigh with the lid on. Heat the dish again in the oven for 30 minutes.

E2.3 Repeat the process until the loss in mass between two successive weightings in less than one milligram. Record the lowest mass obtained.

E2.4 Calculation

Total volatile matter at 105 ^{0}C percent by mass= $\frac{100(M1-M2}{M1-M3}$

Where,

M1 mass in gram of the dish with the material before heating
M2 mass in gram of the dish after heating
M3 mass in gram of the empty dish

Annex F

(normative)

Determination of acid value

F1 Principle

The acid value is determined by titrating the material in benzene-alcohol medium with potassium hydroxide solution:

F2 Reagents

F2.1 Benzene - neutral to phenolphthalein indicator.

F2.2 Rectified Spirit - neutral to phenolphthalein indicator

F2.3 Standard Potassium Hydroxide Solution 0.5 N

F2.4 Phenolphthalein Indicator Solution Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit and dilute with water to 100 ml.

F3 Procedure

F3.1 Mix the material thoroughly, making it entirely liquid before weighing.

F3.2 Weigh accurately about 5 g of the material in a 250-m1 conical flask. Add 75 ml of a mixture of two parts of benzene and one part of rectified spirit.

F3.3 Heat under reflux until the sample is dissolved. Allow it to cool to room temperature and titrate with standard potassium hydroxide solution using phenolphthalein as indicator.

F3.4 Calculation

$$\text{Acid value} = \frac{56.1VN}{M}$$

Where

V volume in ml of standard potassium hydroxide solution used,

N normality of standard potassium hydroxide. Solution, and

M mass in g of the material taken for the test.

Annex G

(normative)

Determination of saponification value

G1 Principles

The material saponified by refluxing with a known excess of alcoholic potassium hydroxide solution. The alkali consumed for saponification is determined by titrating the excess alkali with standard acid.

G2 Apparatus

G2.1 Conical Flasks - 250 to 300 ml capacity made of alkali-resistant glass.
G2.2 Reflux condenser - at least 65 cm long.
G2.3 Balance readable to 1mg
G2.4 Hot plate or water bath

G3 Reagents

G3.1 Methyl Ethyl Ketone
G3.2 Rectified Spirit -
G3.3 Alcoholic Potassium Hydroxide. Solution -Dissolve 30 g of potassium hydroxide in rectified spirit *and* make up to 1 litre. Allow to settle overnight in a dark place, decant the clear liquid and keep in a bottle closed tight with cork or rubber stopper.
G3.4 Phenolphthalein Indicator Solution
G3.5 Standard Hydrochloric Acid 0.5 N.

G4 Procedure

G4.1 Weigh accurately about *2.0* g of the material in a tarred conical flask. Add *25* ml of methyl ethyl ketone, followed by *25* ml of alcoholic potassium hydroxide solution. Add a few pieces of pumice stone and connect the reflux condenser to the flask. Heat the flask on a water-bath or electric hot-plate for about 2 hours. Boil steadily but gently. After the flask and condenser have cooled, wash down the inside of the condenser with about 10 ml of rectified spirit. Add about 1 ml of phenolphthalein indicator solution and titrate with standard hydrochloric acid. Carry out a blank determination at the same time.

G4.2 Calculation

$$\text{Saponification value} = \frac{56.1(B - S)N}{M}$$

Where,

B = volume in ml of standard hydrochloric acid required for the blank,
S = volume in ml of standard hydrochloric acid required for the material,
N =normality of standard hydrochloric acid, and
M = mass in g of the material taken for the test.

Annex H
(normative)

1 SCOPE

This committee draft specifies the method of determing the ester value in bees wax by calculation

2 Expression of result

The ester value shall be calculated as follow:

2.1 The acid value determined according to annex F shall be subtracted from the saponification value determined according to annex G to get the ester value

Annex J
(normative)

Test for fats and fatty acids.

J1 Rageants

J1.1 Sodium Hydroxide Solution - 10 percent.
J1.2 Dilute Hydrochloric Acid - approximately 4N

J2 Procedure

J2.1 Boil 5.0 g of the material for about 10 minutes with 80 ml of sodium hydroxide solution. Replace the water lost by evaporation. Cool and filter the solution through glass wool. Make the filtrate acidic with dilute hydrochloric acid.

J2.2 The material shall be taken to have passed the test if the solution does not become turbid after acidification.

Annex K
(normative)

Test for paraffin and other waxes

K1 Apparatus

K1.1 Balance readable to 0.1g

K1.2 Conical flask 250 ml

K1.3 Reflux condenser

K1.4 Water bath or hot plate

K2 Reagents

K2.1 Alcoholic Potassium Hydroxide Solution approximately 0.5 N, prepared by dissolving potassium hydroxide in 95 percent ethanol

K2.2 *Ethanol 95%*

J3 Procedure

K3.1 Weigh 1.0 g of the material and place it in a conical flask fitted with a water-cooled reflux condenser. Add 10 ml of alcoholic potassium hydroxide solution. Boil under reflux for one hour. Detach the flask from the condenser, insert suitably a thermometer into the liquid in the flask and allow to cool, stirring constantly.

K3.1.1 The material shall be taken to have passed the test if the following conditions are satisfied:
a) The liquid does not become cloudy at a temperature higher than 61°C but becomes cloudy at a temperature between 61°C and 59°C, and

b) Precipitation of large flocks occurs at not more than 2°C below the temperature at which the liquid becomes cloudy.

Example:
If the liquid becomes cloudy at 60°C, precipitation should take place at a